

The 1994 Verification of Soil Remediation (VSR) document should no longer be used unless specifically mandated by a historic settlement document. In the future, the 2002 Sampling Strategies and Statistics Training Materials for Part 201 Evaluations (S3TM) should be referred to for recommendations related to verification of soil remediation. Portions of the VSR have been updated and incorporated into S3TM. Tabbed Section 4.0 of the S3TM should be referred to for recommendations on soil verification sampling and Tabbed Section 7.0 should be referred to for statistical analysis of verification data.

A link to the S3TM is provided below:

<http://www.deq.state.mi.us/documents/deq-erd-stats-s3tm.pdf>



## ~~WASTE MANAGEMENT DIVISION~~

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# ~~GUIDANCE DOCUMENT~~

## ~~VERIFICATION OF SOIL REMEDIATION~~

## ~~ENVIRONMENTAL RESPONSE DIVISION~~

## ~~WASTE MANAGEMENT DIVISION~~

~~APRIL 1994, Revision 1~~

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~~**PLEASE NOTE: STATISTICAL TABLES MAY NOT PRINT COMPLETELY WHEN THIS DOCUMENT IS PRINTED AND SHOULD BE USED AS A REFERENCE ONLY. TO ASSURE THAT THE TABLES ARE ACCURATE, PLEASE REQUEST A PAPER COPY OF THIS DOCUMENT. Additionally, this HTML encoded version of the VSR document will only be accurately displayed with browsers that can support tables, superscripted and subscripted characters (i.e. Internet Explorer 3.0 and Netscape 3.0).**~~

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## ~~EXECUTIVE SUMMARY~~

~~The document provides guidance for sampling soils to verify that soil contamination has been remediated to Type A or Type B criteria in accordance with Act 307 P.A. 1982, as amended. This document is not designed to either guide investigations to determine whether a release has occurred or the nature and extent of an identified release, nor to guide due diligence by a potential property owner. Issuance of this guidance document does not invalidate remedial action plans (RAPs) or clean-ups previously conducted and approved by the DNR.~~

~~Soil sampling and analyses to verify that site remediation is complete can result in two basic errors.~~

- ~~• Declaring a site clean when it is contaminated~~
- ~~• Declaring a site contaminated when it is clean~~

~~A soil sampling plan submitted to the DNR must minimize these errors. The guidance document~~

~~presents acceptable methods for verifying soil remediation. It contains guidance on soil sampling protocols and documentation necessary to characterize and verify cleanup of contaminated soils. The document provides recommended procedures for establishing soil background concentrations, sampling grids, chemical constituent evaluations, statistical comparisons, verifying excavation and in-situ and ex-situ remedies, evaluating treated soils, and soil characterization. The recommended procedures are **not absolutes**. Other methods are available to verify soil remediation. The Department of Natural Resources will evaluate other sampling and statistical strategies on a case-by-case basis.~~

~~The guidance document is divided into two parts:~~

- ~~• **Part 1** contains guidance for small site cleanup verification (less than 10,890 square feet <0.25 acre). It is a "biased" sampling strategy recommending soil sampling from areas most likely to contain contamination.~~
- ~~• **Part 2** contains guidance for soil characterization and cleanup verification of medium and large sites (greater than 10,890 square feet >0.25 acre). It is a statistical random sampling strategy that minimizes biases in sampling.~~

~~Both sampling strategies require discrete soil samples. Compositing samples for cleanup verification is not accepted without prior DNR approval.~~

~~The guidance document contains verification checklists and reporting sections. The reporting sections should be carefully followed in reporting sampling rationale.~~

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~~**Reader's Note:** Questions regarding this guidance document should be directed to Department staff you are currently working with for your project or site.~~

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~~APRIL 94, Revision 1~~

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## ~~**TABLE OF CONTENTS - DNR GUIDANCE DOCUMENT**~~

### ~~**VERIFICATION OF SOIL REMEDIATION**~~

#### ~~**PART 1 - Small Site Soil Cleanup Verification, Introduction 1**~~

~~Verifying Excavation Remedies 2-4~~

~~Sample Locations 2~~

~~Number of Samples 3-4~~

~~*Number of Excavation Floor Samples 3*~~

~~Number of Excavation Sidewall Samples 3-4~~

~~Verifying In-Situ and Ex-Situ Soil Remedies 4~~

~~Sample Analysis 4~~

~~Background Soil Samples 5-8~~

~~Establishing Soil Background 5~~

~~Statistical Analysis for Establishing Background Concentrations 6-8~~

~~1. Mean Plus 3 Standard Deviation Approach 6-7~~

~~2. Other Statistical Procedures for Establishing Background 8~~

~~Procedures for Non-Detect Values 8~~

~~Report for Small Site Verification 9~~

---

## ~~TABLE OF CONTENTS - DNR GUIDANCE DOCUMENT~~

### ~~VERIFICATION OF SOIL REMEDIATION~~

#### ~~PART 2 - Medium and Large Site Soil Cleanup Verification, Introduction 10~~

~~Establishing Grid Interval 11-14~~

~~1. Basic Strategies 11-12~~

~~2. Setting the Grid 12~~

~~3. Variations on Basic Strategy 13-14~~

~~Sampling of Grid 14~~

~~Establishing Soil Background 14-15~~

~~Statistical Analysis for Establishing Background Concentrations 15-18~~

~~1. Mean Plus 3 Standard Deviation Approach 15-17~~

~~2. Tolerance Limit 17~~

~~3. t-tests 18~~

~~4. Other Statistical Procedures for Establishing Background 18~~

~~Procedures for Non-Detect Values 18~~

~~Statistical Evaluation of Data 18~~

~~Grid Approach to Additional Remediation 19~~

~~*Two-Dimensional Node Sampling Excavation Grid 19*~~

~~*Two-Dimensional Subcell Sampling Excavation Grid 19*~~

~~*Three-Dimensional Cleanup Verification 19*~~

~~*Batch Sampling for Ex-Situ Treatment Processes 19*~~

~~Disposal Options 20~~

~~Report for Medium & Large Site Verification 20~~

~~RCRA Clean Closure Certification Checklist 20~~

~~Cited References 42~~

---

## ~~ATTACHMENTS - DNR GUIDANCE DOCUMENT~~

### ~~VERIFICATION OF SOIL REMEDIATION~~

~~attachment attachment page #~~  
~~#~~

~~1 Guide to Sample Bias 21-22~~

~~2 Sampling Protocol for Characterizing~~

~~Waste/Treatment Levels 23-38~~

~~Strategies for Evaluating Treated~~

~~Soils and Waste Materials 23~~

~~Statistical Sampling Strategies 24~~

~~Random Numbers Table 25-27~~

~~Sampling Grids 28~~

~~Statistical Evaluation of Waste/Treatment Characterization Samplings ...29-31~~

~~Example: Calculation of Confidence Limits & Lamda Calculation 32-34~~

~~Table: Cumulative t Distribution 35~~

~~Table: Number of Observations for t test of Mean 36~~

~~Sampling Process Streams 37~~

~~Random Time Waste Sampling Example 38~~

~~3 Tolerance Factors (K): [NTIS Document PB89151047, Table 5] 39~~

~~4 Waste Management Division's Clean Closure Certification Checklist 40-41~~

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## ~~DNR GUIDANCE DOCUMENT, PART 1~~

### ~~SMALL SITE SOIL CLEANUP VERIFICATION (LESS THAN 10,890 SQUARE FEET)~~

~~Part 1 of this document is a guide for a biased sampling strategy to verify that soil contamination has been remediated at sites no greater than 0.25 acres (small sites). Soil sampling and analyses to verify that a site remediation is complete can result in two basic errors:~~

- ~~• Declaring a site clean when it is contaminated~~
- ~~• Declaring a site contaminated when it is clean~~

~~A soil sampling plan submitted to the DNR must minimize these errors. Part 1 presents a biased sampling method of verifying soil remediation at small sites. The biased sampling approach specified in this guidance recommends soil sampling from areas most likely to still exceed cleanup criteria. The location of the soil sample points relies on a site specific analysis of the released or contaminant distribution and the soil types encountered. The remediation is verified using a point by point comparison of sample values with the appropriate cleanup criteria. If the cleanup criteria are exceeded at any sampled point, the biased sampling methodology may require additional remediation at that point until the criteria are met. Verification of cleanup utilizing the biased approach should generally require fewer samples to demonstrate attainment than by using the unbiased approach. DNR will evaluate other sampling and statistical strategies on a case-by-case basis.~~

~~Any biased sampling plan, whether presented in the guidance document or some other geostatistical approach, requires professional judgment. Therefore, documentation and the rationale used to select sample locations are extremely important. The report section (page 9) of this guidance document should be carefully followed.~~

~~Compositing samples for verifying soil remediation is not acceptable without prior DNR approval. When verifying a soil remediation is complete, contaminant concentrations will be low. Compositing may result in the contaminant concentrations not being representative of what remains in the soil. If concentrations are low, compositing may dilute the concentrations of a contaminant to below its threshold detection limit. Additionally, if contamination is indicated in a composited sample, the location of the contamination remains unknown.~~

~~Part 1 is divided into five main sections: Verifying Excavation Remedies, Verifying In-Situ and Ex-Situ Soil Remedies, Sample Analysis, Background Soil Samples, and Reports. The excavation and in situ remediations require different strategies for verification. Guidance is presented for statistically determining background concentrations of compounds/contaminants. Guidance for reporting all appropriate information is presented to facilitate remediation approval.~~

~~-1-~~

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## ~~VERIFYING EXCAVATION REMEDIES~~

~~Verifying that contaminated soil is remediated by means of excavation requires samples from the excavation bottom and sidewalls. Tables and formulas presented provide the minimum number of samples necessary to verify cleanup for various size excavations. The biased approach specified in this guidance recommends soil sampling from areas most likely to still exceed cleanup criteria. The location of the sample collection points relies on site specific analysis of the release or contaminant distribution and the soil types encountered in the excavation. The minimum number of excavation floor and sidewall samples required to demonstrate verification using a point by point comparison with the cleanup criteria are specified. If the cleanup criteria are exceeded at any point, this verification methodology may require additional excavation at that point until the criteria are attained.~~

~~Sampling and analyzing the locations most likely to have contaminants can minimize the number of samples needed to verify remediation is complete. Since professional judgment and site specific knowledge are required for selecting sampling locations, the rationale used to select these locations must be documented in the verification report.~~

## ~~SAMPLE LOCATIONS~~

~~Using a biased sampling approach, samples must be collected where they will most likely encounter contamination which could exceed the cleanup criteria. This will minimize the number of samples needed to verify a site is remediated. A sampling strategy that uses bias to choose sample locations is recommended. While it is inappropriate for this guidance document to dictate exact locations for sample collection in this strategy, site specific information (e.g., the location of leaks in an underground storage tank or its piping) from the remedial investigation concerning the release and soil conditions should be used along with professional judgment and the general guidance provided here to select appropriate soil sampling locations.~~

~~EXAMPLE: It would be incorrect to sample the north side of an excavation pit as extensively as the south side when the leak was confirmed on the south side of the tank.~~

~~Because a site must be remediated to a certain degree before approval can be considered, an analysis of data generated by a prior investigation should yield information for the verification analysis. The field personnel present during remediation should be sufficiently familiar with the conditions on-site to implement an appropriate verification strategy. A soil verification strategy should incorporate all pertinent biases of a site which may include, but are not limited to, those listed below.~~

- ~~• preferential pathways of contaminant migration~~
- ~~• source areas~~
- ~~• stained soils~~

- other site specific "clues" (e.g., fractures in clays)
- changes in soil characteristics (e.g., sand/clay interfaces)
- soil types and characteristics

-2-

## NUMBER OF SAMPLES

The following tables are used to determine the minimum number of samples necessary from the floor and sidewalls of an excavation no greater than 0.25 acres using a biased sampling approach. If the area of the excavation floor exceeds 10,890 square feet, use Part 2 of this guidance document. A site may have an appropriate number of samples collected for verification, but, if the samples are not collected from the appropriate locations (discussed previously) and adequately reported, remediation may not be considered adequate. All sample locations must be accurately located, described, and reported. It should be noted that "excavation" as used here refers only to that area excavated for remediation purposes and being verified to meet Type A/Type B cleanup criteria.

### *Number of Excavation Floor Samples*

Determine the minimum number of excavation floor samples from the table below.

<b>TABLE 1</b>	
<b>EXCAVATION FLOOR SAMPLES</b>	
<i>Area of Floor (sq ft)</i>	<i>Number of Samples</i>
< 500	2
500 < 1,000	3
1,000 < 1,500	4
1,500 < 2,500	5
2,500 < 4,000	6
4,000 < 6,000	7
6,000 < 8,500	8
8,500 < 10,890	9

### *Number of Excavation Sidewall Samples*

Sidewall samples are required to verify that the horizontal extent of contamination has been remediated. Use Table 2 to determine the minimum number of required sidewall samples. In no case is less than one sample on each sidewall (i.e., four) acceptable. In the case of irregularly shaped excavations, where four walls are not readily discernible, divide the total wall area into four segments of approximately equal size. Sidewall samples should be located in accordance with "biases" outlined earlier in Part 1.

-3-



**TABLE 2****EXCAVATION SIDEWALL SAMPLES**

<i>Total Area of Sidewalls (sq ft)</i>	<i>Number of Samples</i>
< 500	4
500 < 1,000	5
1,000 < 1,500	6
1,500 < 2,000	7
2,000 < 3,000	8
3,000 < 4,000	9
> 4,000	1 sample per 45 lineal feet of sidewall

**VERIFYING IN-SITU AND EX-SITU SOIL REMEDIES**

The effectiveness of in-situ soil remedies must be verified by three-dimensional random soil sampling. Refer to Attachment 2 for approved statistical sampling strategies. Certain ex-situ remedies, such as bio-piles or above-ground vapor extraction, may be amenable to statistical sampling strategies or batch sampling. Any proposed sampling strategy for in-situ or ex-situ remedies should be pre-approved by the DNR.

**SAMPLE ANALYSIS**

All test methods and associated target detection levels for cleanup verification must be consistent with those specified in MERA Operational Memorandum #6. Also, MERA Operational Memorandum #13 may be reviewed to evaluate appropriate QA/QC procedures. Generally, constituents in soil will be measured on a total, dry weight basis.

**BACKGROUND SOIL SAMPLES****ESTABLISHING SOIL BACKGROUND**

Establishing soil background, as required by Act 307 PA 1982, as amended, Michigan Environmental Response Act (MERA), can be accomplished by utilizing Operational Memorandum #15 or using the following guidance:

Background should be established as appropriate for site specific waste constituents, specific chemicals used in various processes, facility operations, or remedial investigation results. Sample analyses may include metals, organic constituents, or other site specific waste constituents. Analyses should be in accordance with MERA Operational Memorandum #6




Many factors can play a part in the background concentrations of a chemical in soil.

EXAMPLE: The geologic origin (e.g., the parent rock) of glacial drift may have been high in copper, lead, or other metals that may be potential contaminants. Additionally, the hydrogeologic situation can

~~alter the quantity of these elements. Groundwater recharge areas (e.g., highlands) are frequently leached of metals while groundwater discharge areas (e.g., swamps, floodplain) are the recipients of leached metals. Thus, sites in low areas will usually have higher background concentrations than upland areas. Other conditions, such as precipitation and atmospheric fallout from widely dispersed human and natural activities, also affect soil concentrations.~~

~~A minimum of four samples must be used to establish "background" in soils. This will help account for natural constituent occurrences and inherent variability within each distinctive soil horizon. Background samples must be collected in an area which has not been impacted by environmental contamination from the site and representative of natural background conditions. Based on waste type, contaminant mobility, operation practices, and soil type (sand, silty sand, clay), an estimate of contamination depth should be made and background samples taken at comparable depths for the particular soil type. Multiple soil horizons should have "background" established separately (e.g., minimum of four samples per each soil unit).~~

~~EXAMPLE:~~

<b>Ground Surface</b>		
<del>Brown medium-coarse SAND</del>		<del>4 samples</del>
<del>Lt. brown silty fine SAND</del>		<del>4 samples</del>
<del>Gray silty CLAY w/trace of fine-med sand</del>		<del>4 samples</del>

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## ~~STATISTICAL ANALYSIS FOR ESTABLISHING BACKGROUND CONCENTRATIONS~~

~~The recommended statistical method for establishing background concentrations at small sites is (1) establishing the upper limit of background concentration of a constituent at the mean plus 3 standard deviations or (2) other statistical methods submitted to DNR for approval.~~

### ~~1. Mean Plus 3 Standard Deviation Approach~~

~~Calculate the "upper limit" of background concentration by using the following 5 step process:~~

~~A. Calculate the background mean ( $\bar{X}_p$ ) by dividing the sum of the total background readings by the total number of background readings:~~

$$\bar{X}_b = (X_1 + X_2 + \dots + X_n) / n$$

B. Calculate the background variance ( $S_b^2$ ) by taking the sum of the squares of each reading minus the mean and dividing by the degrees of freedom (the total number of background samples minus one):

$$S_b^2 = ((X_1 - \bar{X}_b)^2 + (X_2 - \bar{X}_b)^2 + \dots + (X_n - \bar{X}_b)^2) / n - 1$$

**NOTE:** Any sample populations less than (n < 30 samples) must use n - 1 for degrees of freedom

C. Calculate the background standard deviation ( $S_b$ ) by taking the square root of the variance:

$$S_b = (S_b^2)^{1/2}$$

D. The Coefficient of Variation Test (CV) where

$$CV = S_b / \bar{X}_b$$

is used to evaluate data distribution. The background data should generally have a CV of less than 0.5 for granular soils, less than 0.75 for cohesive soils, or an explanation accounting for higher CV values. The maximum recommended CV is 1. If the data distribution exceeds a CV of 1.0, then a thorough evaluation will need to be made to account for this variability (e.g., lab QA/QC, typographical errors, soil classification, sample location, data not normally distributed, etc.). If the CV exceeds 1.0 and there is sufficient evidence to suggest a data point does not accurately represent background conditions or if QA/QC problems exist which invalidate that data point, the outlier data may be dropped or additional samples collected and analyzed to ensure a sufficient representative data population (n) is achieved. A high concentration in and of itself is not sufficient justification to exclude the data point.

E. Use the  $\bar{X}_b + 3 \cdot S_b$  of "background" data as the maximum allowable limit or upper limit.

Where  $3 \cdot S_b$  equals three times the standard deviation and  $\bar{X}_b$  equals the background mean (this statistical method only requires one sample per station). Compare each sample point to the calculated maximum allowable limit or upper limit analyzed from background data.

**EXAMPLE:** Four sand samples from a site were analyzed for background concentrations for lead. Concentrations of lead from the sample analyses returned from the lab were 56, 25, 18, and 35 ppb. Now, the investigator wants to examine the data set to discover whether the 56 ppb sample is an outlier:

$$\bar{X}_b \text{ (mean)} = (56 + 25 + 18 + 35) / 4 = 33.5$$

$$S_b^2 = ([56 - 33.5]^2 + [25 - 33.5]^2 + [18 - 33.5]^2 + [35 - 33.5]^2) / 3 =$$

$$S_b = (\text{standard deviation}) = (S_b^2)^{1/2} = 16.5$$

$$CV = 16.5/33.5 = 0.49$$

Because 0.49 is less than 0.5, no further evaluation of the background data set is necessary.

Therefore, the background upper limit value for this site is:

$$\bar{x}_b + 3s_b = 33.5 + (3 * 16.5) = 83.0 \text{ ppb}$$

If a value is found to be an outlier which is not representative of background conditions, it may be replaced by another sample that is not an outlier to maintain at least four samples for background determination.

-7-

2. *Other statistical procedures for establishing background.* Refer to a statistical reference book or US EPA's Interim Final Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities (April 1989) and Addendum (July 1992).

## PROCEDURES FOR NON-DETECT VALUES

The following provides some guidelines in incorporating non-detectable sample results into the procedure to calculate background concentrations.

1. If less than 50% of the background data is below the detection limit (DL), use 1/2 of the detection limit as the value.
2. If more than 50% of the background data is below the detection limit, use one of the following procedures:
  - Alternate "0" and the detection limit (DL) resulting in a net value of of the detection limit with a variance.

EXAMPLE:	Actual Value	Substitute Value
	<DL	DL
	<DL	0
	<DL	DL
	<DL	0

- The Continuity Correction procedure with the t-test, Cohen's method, or other approved methods.

-8-

## ~~REPORT FOR SMALL SITE VERIFICATION~~

~~Soil cleanup verification reports for small sites must identify the number and location of samples and justify the sample location selected (why and how). The verification report must include the following.~~

### ~~1. MAP(s) and CROSS SECTIONS~~

~~Provide a sealed map of the floor and walls of an excavation (the vertical and horizontal area treated for in-situ remediations) with sample locations identified. The cross section should depict the stratigraphy, fractures, soil types, discolorations, unusual characteristics, odor, etc.~~

### ~~2. SAMPLE LOCATION RATIONALE~~

~~A. Background sample locations~~

~~B. Verification sample locations~~

~~C. Sample depths~~

~~D. Sample collection procedures~~

~~E. Describe biases and rationale used for collecting each sample (e.g., clay fractures, discolored soil, location of leak in tank)~~

### ~~3. DATA ANALYSES~~

~~A. Analytical parameters~~

~~B. Analytical methods used~~

~~C. Method detection limits~~

~~D. Laboratory Quality Assurance/Quality Control~~

### ~~4. STATISTICAL ANALYSES~~

~~A. Calculation of background concentrations~~

~~B. Coefficient of variance calculations~~

~~C. Lab results~~

~~D. Narrative explanation of background concentrations~~

~~-9-~~

## ~~DNR-GUIDANCE DOCUMENT, PART 2~~

### ~~MEDIUM AND LARGE SITE SOIL CLEANUP VERIFICATION~~

#### ~~(GREATER THAN 10,890 SQUARE FEET)~~

~~Part 2 describes statistical random sampling strategies to verify the remediation of medium and large sites greater than 0.25 acres in size. The strategies employ the use of gridding to facilitate the unbiased selection of sampling points and accepted statistical tools for evaluating the resultant data. The strategies~~

~~provide a 95% confidence level of determining any hot spot concentrations on a site. It contains guidance on sampling protocol and necessary documentation for clean closures. Part 2 also discusses how to establish grid intervals, set grids, sample grids, statistically evaluate the data, use grids to guide additional remedial activities, disposal options, reporting, and a certification checklist. It also provides guidance on the sampling of ex-situ remedial processes (e.g., thermal desorption).~~

~~The term 'clean closure' means that the site has been restored to either Type A or Type B levels. Type A is defined in Act 307 P.A. 1982, as amended, which references nondetect or background levels. Type B is defined in Act 307 P.A. 1982, as amended, which references riskbased or background levels. Waste, soil, other environmental media, and/or debris removed should be classified as hazardous or non-hazardous to determine disposal options and handling requirements (i.e., solid waste under Act 641 P.A. 1978, as amended; hazardous waste under Act 64 P.A. 1979, as amended; land ban restrictions under 40 CFR Part 268).~~

~~All cleanup verification evaluations must consider the spatial arrangements of sample values (patterned vs totally random) and the impacts on the present and future uses of the site. Because Type B cleanups are based on residual risk, the distribution of that risk, now and in the future, must be determined. These procedures are **not absolutes**. Other sampling approaches may be developed and submitted for DNR approval.~~

~~Three of the statistical sampling strategies most commonly used for evaluating remedial sites and wastes are described in Attachment 2. For further discussion on sampling strategies and sample collection methods, see "Test Methods for Evaluating Solid Waste," SW-846 Volume II: Field Methods, November 1986, Third Edition, US EPA.~~

~~Compositing samples for verifying soil remediation is not acceptable without prior DNR approval. When verifying a soil remediation is complete, contaminant concentrations will be low. Compositing may result in the contaminant concentrations not being representative of what remains in the soil. If concentrations are low, compositing may dilute the concentrations of a contaminant to below its threshold detection limit. Additionally, if contamination is indicated in a composited sample, the location of the contamination remains unknown.~~

~~-10-~~

## ~~ESTABLISHING GRID INTERVALS~~

~~When obtaining samples to verify that soil or wastes have been adequately remediated, it is important to insure that the analytical results obtained will provide an accurate representation of the entire area or volume under consideration. The location and number of samples to be taken at a particular remediation site depends on many factors: the level of confidence desired, the spatial and temporal variability of the media to be sampled, and the costs involved. An important objective in any sampling program is to obtain the most accurate data possible while minimizing the associated costs. One method to accomplish this goal is to use statistically valid sampling strategies. The appropriate sample number can be estimated and the sampling locations can be chosen without bias.~~

~~Such strategies employ the use of gridding to facilitate the unbiased selection of sampling points and accepted statistical tools for evaluating the resultant data. Statistical theory allows for the sampling of a subset of the grid points to achieve a reliable characterization of the entire remedial area or waste. Subsections describe ways to use sampling grids and statistical tools to evaluate areas of remediation.~~

~~The following equations and tables provide a simple basis to establish a grid system to facilitate unbiased selection of sampling points and sample coverage proportional to the area being verified.~~

~~1. *Basic Strategies.* A grid system should be established over the area being remediated. Grid point representation should be proportional to the size of the area. For excavation, both the sidewalls and bottom areas would be included in the determination of the area size. It is recommended that one of the following equations be used to determine grid intervals for stationing:~~

~~small site: see Part 1~~

$$\text{medium site: } (A/\pi)^{1/2} / 4 = GI$$

$$\text{large site: } ((A * \pi) / SF)^{1/2} = GI$$

~~WHERE:      A = area to be grid (square feet)~~

~~GI = grid interval~~

~~SF = Site Factor, length of area to be grid (unitless)~~

~~pi = 3.14159~~

~~-||-~~

~~It appears that there are logical size ranges of sites to which the grid equations apply:~~

~~A) small: up to 0.25 acre~~

~~B) medium: 0.25-3.0 acres~~

~~C) large: 3.0 acres and greater~~

~~To simplify this application, use the following chart based on an average size range of sites (1 acre = 43,560 square feet). The approximate grid ranges are provided as a quick check on numbers generated for specific sites using the above formulas.~~

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<del>Site Acreage*</del>	<del>Square Feet*</del>	<del>Grid Interval Ranges</del>
<del>up to 0.25 (small)</del>	<del>up to 10,890</del>	<del>See Part 1</del>
<del>0.25-3.00 (medium)</del>	<del>10,890-130,680</del>	<del>15-50 feet</del>
<del>3.0 and over (large)</del>	<del>130,680 +</del>	<del>30 feet plus</del>

~~\* Site acreage, square footage, is total area of sidewalls and base of excavation.~~

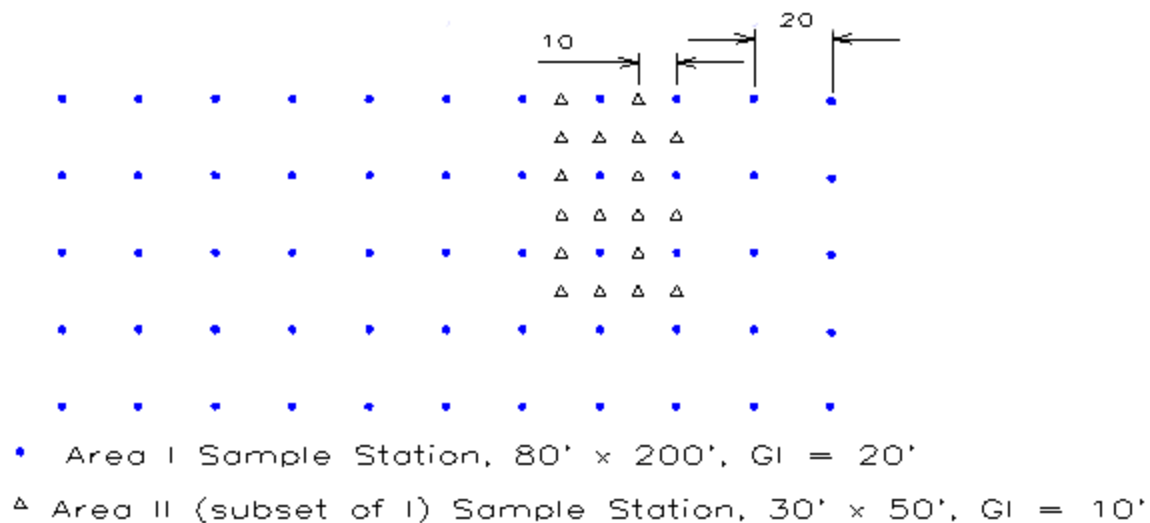
~~2. *Setting the Grid.* After the grid interval is calculated, it is recommended that a scaled grid overlay be~~

made to superimpose on a map of the remediated area (this area includes both sidewalls and base). Some specified point (usually the southwest corner) should be designated as the 0,0 coordinate. The grid can then be adjusted to maximize sampling coverage. Some grid adjustment may be necessary for unusually shaped areas. Grid adjustment may also be needed to accommodate a minimum of at least one sample from each sidewall. Proposals for different grid strategies may be submitted for DNR review and approval on a case-by-case basis.

-12-

### 3. *Variations on Basic Strategy.*

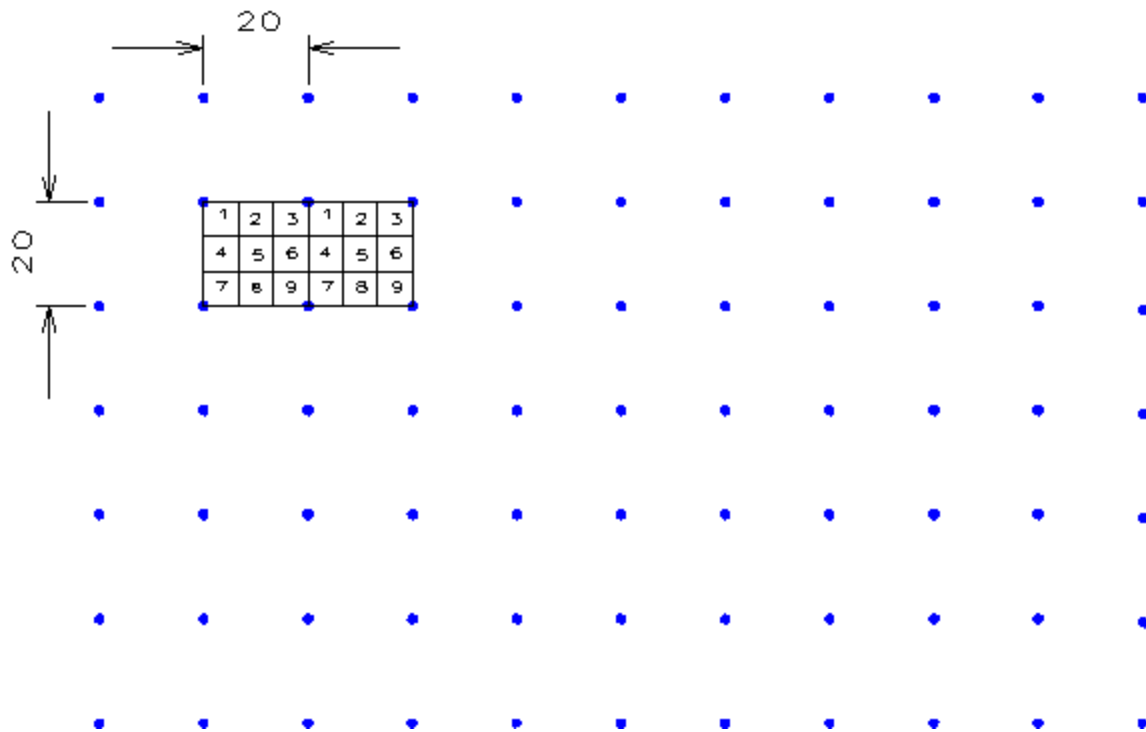
A. Subgridding. It may be warranted to apply grids with different intervals within the remediated area so that a proportional sampling can be focused on suspect areas (such as sumps, tank leak areas, etc.).



B. Further Randomization. Sites that may have a patterned distribution of waste or contamination due to time sequence of filling, production sequences, or physical site conditions (i.e., furrows) may require a further randomization of sampling. In such cases, the following grid cell sampling format may be selected instead of at grid point stations. Each grid cell to be sampled must be divided into nine equal sized "subcells." Next, a random number table is used to select in which of the subcells the sample will be taken. The random number table is used again to select which subcell for the next cell and so on.

EXAMPLE:-





Area = 120' x 200', GI = 20'

In the example above, a sampling grid has been set up with grid point stations 20 feet apart using the appropriate formula. Two cells which have been selected at random have been divided into nine subcells each. Subcell #4 was chosen randomly in one cell and subcell #2 in the other cell. This process is continued for all of the cells selected at random for sampling. Samples are then taken in each randomly chosen subcell.

-13-

### C. Three dimensional gridding: In-Situ and Ex-Situ Remediations.

In-situ and ex-situ remediations involving soils and/or wastes with a significant vertical component should be evaluated in three dimensions (volume evaluation). Examples of such remediations would be in-situ soil vapor extraction or ex-situ bioremediation involving several cubic feet of soil and/or waste. A grid is superimposed on the remediation area as described in the previous sections and a vertical component is added at each node. The vertical sampling increments would be site specific and require prior approval from the DNR. Refer to Attachment 1 "Guide to Sample Bias" for additional guidance on vertical sampling increments.

### SAMPLING OF GRID

Sampling of grids may include all of the grid stations or a phased subset of the total stations. The subset of grid stations is created by assigning coordinates to all the nodes and randomly selecting nodes using a random number generator or a random number table (refer to Attachment 2). A minimum of 12 samples or 25%, whichever is larger, of the total grid stations should be sampled and analyzed initially to allow a large enough data pool for statistical analysis. It is advisable that extra samples also be taken and kept

~~under proper chain of custody and storage procedures at the time of initial sampling. If the statistical analysis indicates that more samples are needed, an additional sample trip to the field may have been avoided. A method for calculating the sample size requirements is presented in Attachment 2 (Lamda relationship).~~

## ~~ESTABLISHING SOIL BACKGROUND~~

~~Establishing soil background, as required by Act 307 PA 1982, as amended, Michigan Environmental Response Act (MERA), can be accomplished by utilizing Operational Memorandum #15 or using the following guidance.~~

~~Background should be established for site specific waste constituents, specific chemicals used in various processes, facility operations, or remedial investigation results. Sample analyses may include metals, organic constituents, or other site specific waste constituents. Analyses should be in accordance with Act 307 P.A. 1982, as amended.~~


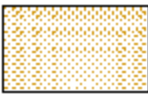
~~Many factors can play a part in the background concentrations of a chemical in soil.~~

~~EXAMPLE: The geologic origin (e.g., the parent rock) of glacial drift may have been high in copper, lead, or other metals that may be potential contaminants. Additionally, the hydrogeologic situation can alter the quantity of these elements. Groundwater recharge areas (e.g., highlands) are frequently leached of metals while groundwater discharge areas (e.g., swamps, floodplain) are the recipients of leached metals. Thus, sites in low areas will usually have higher background concentrations than upland areas. Other conditions, such as precipitation and atmospheric fallout from widely dispersed human and natural activities, also affect soil concentrations.~~

~~-14-~~

~~A minimum of four samples must be used to establish "background" in soils. This will help account for natural constituent occurrences and inherent variability within each distinctive soil horizon. Background samples must be collected in an area which has not been impacted by environmental contamination from the site and representative of natural background conditions. Based on waste type, contaminant mobility, operation practices, and soil type (sand, silty sand, clay), an estimate of contamination depth should be made and background samples taken at comparable depths for the particular soil type. Multiple soil horizons should have "background" established separately (e.g., minimum of four samples per each soil unit).~~

~~EXAMPLE:~~

<b>Ground Surface</b>		
<del>Brown medium-coarse SAND</del>		<del>4 samples</del>
<del>Lt. brown silty fine SAND</del>		<del>4 samples</del>

Gray silty CLAY w/trace of fine med sand		4 samples
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## **STATISTICAL ANALYSIS FOR ESTABLISHING BACKGROUND CONCENTRATIONS**

The recommended statistical method(s) for establishing background concentrations at medium and large sites are (1) establishing the upper limit of background concentration of a constituent at the mean plus 3 standard deviations, (2) tolerance limit, (3) t-tests, and (4) other statistical methods submitted to the DNR for approval.

### **1. Mean Plus 3 Standard Deviation Approach**

Calculate the "upper limit" of background concentration by using the following 5 step process:

A. Calculate the background mean ( $\bar{X}_b$ ) by dividing the sum of the total background readings by the total number of background readings:

$$\bar{X}_b = (X_1 + X_2 + \dots + X_n) / n$$

B. Calculate the background variance ( $S_b^2$ ) by taking the sum of the squares of each reading minus the mean and dividing by the degrees of freedom (the total number of background samples minus one):

-15-

$$S_b^2 = ((X_1 - \bar{X}_b)^2 + (X_2 - \bar{X}_b)^2 + \dots + (X_n - \bar{X}_b)^2) / n - 1$$

**NOTE:** Any sample populations less than (n < 30 samples) must use n - 1 for degrees of freedom

C. Calculate the background standard deviation ( $S_b$ ) by taking the square root of the variance:

$$S_b = (S_b^2)^{1/2}$$

D. The Coefficient of Variation Test (CV) where

$$CV = S_b / \bar{X}_b$$

is used to evaluate data distribution. The background data should generally have a CV of less than 0.5 for granular soils, less than 0.75 for cohesive soils, or an explanation accounting for higher CV values. The maximum recommended CV is 1. If the data distribution exceeds a CV of 1.0, then a thorough evaluation will need to be made to account for this variability (e.g., lab QA/QC, typographical errors,

soil classification, sample location, data not normally distributed, etc.). If the CV exceeds 1.0 and there is sufficient evidence to suggest a data point does not accurately represent background conditions or if QA/QC problems exist which invalidate that data point, the outlier data may be dropped or additional samples collected and analyzed to ensure a sufficient representative data population (n) is achieved. A high concentration in and of itself is not sufficient justification to exclude the data point.

-16-

E. Use the  $\bar{X}_b + 3 \cdot S_b$  of "background" data as the maximum allowable limit or upper limit.

Where  $3 \cdot S_b$  equals three times the standard deviation and  $\bar{X}_b$  equals the background mean (this statistical method only requires one sample per station). Compare each sample point to the calculated maximum allowable limit or upper limit analyzed from background data.

EXAMPLE: Four sand samples from a site were analyzed for background concentrations for lead. Concentrations of lead from the sample analyses returned from the lab were 56, 25, 18, and 35 ppb. Now, the investigator wants to examine the data set to discover whether the 56 ppb sample is an outlier:

$$\bar{X}_b \text{ (mean)} = (56 + 25 + 18 + 35) / 4 = 33.5$$

$$s_b^2 = ([56 - 33.5]^2 + [25 - 33.5]^2 + [18 - 33.5]^2 + [35 - 33.5]^2) / 3 =$$

$$s_b = (\text{standard deviation}) = (s_b^2)^{1/2} = 16.5$$

$$CV = 16.5 / 33.5 = 0.49$$

Because 0.49 is less than 0.5, no further evaluation of the background data set is necessary. Therefore, the background upper limit value for this site is:

$$\bar{X}_b + 3 \cdot S_b = 33.5 + (3 \cdot 16.5) = 83.0 \text{ ppb}$$

If a value is found to be an outlier which is not representative of background conditions, it may be replaced by another sample that is not an outlier to maintain at least four samples for background determination.

2. *Tolerance Limit.* This statistical procedure is a fairly sensitive program for environmental purposes. It minimizes false positive and is simple to perform. **A minimum background data base of n=8** (optimum n=16) is needed for this method. Other suggested criteria follow:

A. The Coefficient of Variation Test (CV) to evaluate data distribution. See this Guidance Document, Part 2, Statistical Analysis for Establishing Background Concentrations, #1.D. (the Coefficient of Variation Test...).

B. Using the mean ( $\bar{X}_b$ ) and standard deviation ( $S_b$ ), construct the onesided upper tolerance limit (TL) by taking the mean plus a tolerance coefficient (K) at the 95% probability level for a 95% coverage (for

K values, see Attachment 3) times the standard deviation as follows:

$$TL = \bar{X}_b + KS_b$$

-17-

3. *t*-tests. Any test should be "approved" by DNR prior to use since there are a number of variations. The Gosset Student *t*-test (1908) or Cochran's Approximation to the Behren's Fisher Student's *t*-test as referenced in the 40 CFR Part 264, Appendix IV, are recommended. Note that these statistical comparison methods require that two or more discrete samples be taken at each sampling station.

4. *Other statistical procedures for establishing background.* Refer to a statistical reference book or US EPA's Interim Final Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities (April 1989) and Addendum (July 1992).

## PROCEDURES FOR NON-DETECT VALUES

The following provides some guidelines in incorporating non-detectable sample results into the procedure to calculate background concentrations:

1. If less than 50% of the background data is below the detection limit (DL), use 1/2 of the detection limit as the value.
2. If more than 50% of the background data is below the detection limit, use one of the following procedures:
  - Alternate "0" and the detection limit (DL) resulting in a net value of of the detection limit, with a variance

<del>EXAMPLE:</del>	<del>Actual Value</del>	<del>Substitute Value</del>
	<DL	DL
	<DL	0
	<DL	DL
	<DL	0

- The Continuity Correction procedure with the *t*-test, Cohen's method, or other approved methods.

## STATISTICAL EVALUATION OF DATA

A detailed description of an acceptable approach for evaluating the data generated by statistically based random sampling strategies such as those described in the foregoing sections is provided in Attachment 2 (page 29). The 95% upper confidence limit (UCL) of the mean is calculated for each constituent of concern and compared to the regulatory threshold (RT) (i.e., cleanup criterion; e.g., Type A or B). If the

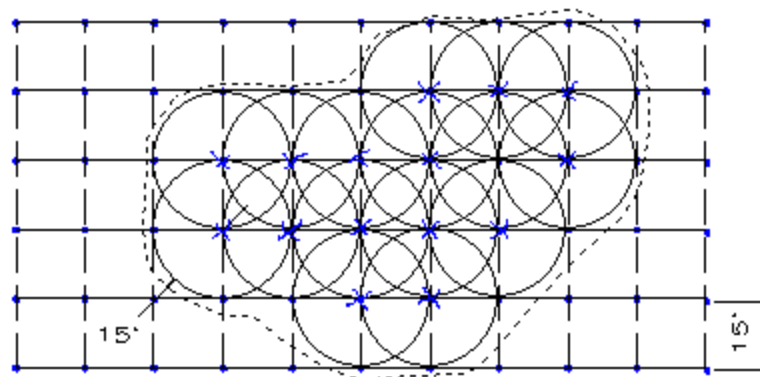
UCL is less than the RT and an adequate number of samples have been collected and spatially evaluated, the remediation is deemed complete. Attachment 2 also provides a step wise procedure for determining whether an adequate number of samples have been collected, based on the analytical data derived from the initial and subsequent rounds of samples. **All evaluations must consider the spatial correlation of sample values** (e.g., highest concentrations in the same area), **present and future uses of the site, residual risk, and distribution of that risk now and in the future.** Other acceptable methods for UCL and sample size calculations can be found in US EPA SW-846, Third Edition, Section 9.1.1.3.

-18-

## GRID APPROACH TO ADDITIONAL REMEDIATION

1. *TwoDimensional Node Sampling Excavation Grid.* Verification sampling as described above will at times indicate that remediation is incomplete. Excavation of contaminated areas should be based on the established grid system interval (as recommended in this Guidance Document, Part 2). Where a subset of grid points has indicated that the entire area exceeds the cleanup, the nodes adjacent to the sampled nodes that are causing the exceedance should be sampled, and this process repeated until the "Hot spots" requiring removal have been defined. The radius of excavation around the contaminated sample point(s) is equal to the grid interval (GI=r). Excavation depth is to the deepest point of contamination or to the depth where acceptable levels are anticipated. After excavation, the impacted point(s) must be resampled at their new elevations to verify that the area meets the selected cleanup criteria. If continued contamination is detected, the excavation format is repeated until a satisfactory result is obtained.

### EXAMPLE:



GL = 150

A = 11,250

GI = 14.9

• Sample Station

x Contaminated Station

r = GI = 15 feet

~~Remediation of contaminated soil by excavation will be in accordance with Act 307 P.A. 1982, as amended. The proposed remedial action plan must be approved by the DNR.~~

~~2. *Two-Dimensional Subcell Sampling Excavation Grid.* Use this Guidance Document, Part 2. The radius of excavation around a contaminated point may need to be adjusted to greater than the GI distance. This adjustment is due to the variable distances between sampling points.~~

~~3. *Three-Dimensional Cleanup Verification.* If sampling and statistical analysis using this Guidance Document indicate that Act 307 cleanup criteria have not been met, additional remediation will be required. The sampling protocol and strategies described in Attachment 2 and in SW-846, Third Edition, Volume II, Part III, Chapter 9, are acceptable. All sampling strategies, detection levels, and sampling pathways must be in accordance with Act 307 P.A. 1982, as amended. If any portion of the soil mass in question appears to be causing the material to fail, it may be identified through hot spot sampling and selectively removed. Subsequent sampling must be done to confirm that the remaining material meets Act 307 P.A. 1982, as amended.~~

~~4. *Batch Sampling for exsitu treatment processes.* If exsitu treatment processes of contaminated soil or waste is used in the remediation, a sampling program for the process stream needs to be developed. The basis of this program is to get representative samples over time versus a spatial approach (Attachment 2, Sampling Process Streams).~~

~~-19-~~

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## ~~DISPOSAL OPTIONS~~

~~Soils remediated to Act 307 P.A. 1982, as amended, standards (Type A and/or Type B) are no longer considered a waste per Act 64 P.A. of 1979, as amended, and RCRA regulations. Disposal of excavated waste, soil, other environmental media, and/or debris must be in accordance with all applicable Federal, State, and local regulations.~~

## ~~REPORT FOR MEDIUM AND LARGE SITES VERIFICATION~~

~~Soil cleanup verification reports for medium and large sites must identify the number and location of samples and justify the sample location selected (why and how). The verification report must include the following:~~

### ~~1. MAP(S) AND CROSS SECTIONS~~

~~Provide a scaled map of the floor and walls of the excavation (the vertical and horizontal area treated for in-situ remediations) with sample grid and sample locations identified. Appropriate cross section should depict the stratigraphy, fractures, soil types, and final depth and elevations of the excavation.~~

### ~~2. SAMPLE LOCATION RATIONALE~~

- ~~a. Properly labeled and easily identified sampling grid stations (map) including background stations~~
- ~~b. Sample Depths~~

~~c. Sample Collection Procedures~~

~~d. Results of all tests to determine clean closure (charts, tables, lab sheets, field notes, well logs, boring logs)~~

### ~~3. DATA ANALYSES~~

~~a. Analytical parameters~~

~~b. Analytical methods used~~

~~c. Method detection limits~~

~~d. Laboratory Quality Assurance/Quality Control~~

### ~~4. STATISTICAL ANALYSES~~

~~a. Explanation and calculation of background concentrations~~

~~b. Statistical comparisons on sampling results compared to background (this should include full computations on background and statistical analysis)~~

~~c. Lab results~~

~~5. Additional information to support closure (e.g., residual risks, spatial correlation of sample values, present and future land uses).~~

## ~~RCRA CLEAN CLOSURE CERTIFICATION CHECKLIST~~

~~Attachment 4 is a guide that indicates the information that a facility should provide to certify that their activities meet the conditions for a clean closure under the Act 64/RCRA regulations.~~

~~-20-~~

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## ~~A T T A C H M E N T 1~~

### ~~GUIDE TO SAMPLE BIAS~~

~~Many factors can play a part in the concentrations of contaminants. The following contains some of the factors impacting chemical concentrations and locations.~~

### ~~CHEMICAL TRANSFORMATIONS~~

~~Many organic chemicals may undergo aerobic and anaerobic degradation. A description of these processes is beyond the scope of this document. The subject is approached here, however, to be sure that samplers are aware that the chemical(s) spilled may not be the only chemical(s) in the soil after a transformation has occurred. These occurrences should be documented in the remedial investigation. The full scan of chemicals from the remedial investigation requiring cleanup should be analyzed when doing a closure. Analyses should be done for all chemicals that have been identified as breakdown products of the chemicals found on-site.~~

~~The professional literature contains many articles on this subject (Cline and Brown, 1989; Borden and Bedient, 1987; Wilson and Wilson, 1985). The interested reader is directed to these articles.~~



### ***Organic Carbon Content of Soil***

The organic carbon content of soils is a key factor in the ability of any soil to adsorb contaminants. For a variety of reasons (Lindsay, 1979), an increase in organic carbon content leads to an increase in the adsorption of several classes of chemicals.

**Where to sample:** Areas of the excavation that appear to have excess organic carbon (e.g. peat, muck, darker soils) should be preferentially sampled.

### ***Medium Sand or Larger Grains***

Medium to larger grain size sand has from 20 to 40 percent porosity. Most sands in Michigan are composed of quartz, limestone, and small amounts of metamorphic rock fragments. These soils have a low capacity for adsorbing metals or hydrophilic (soluble) organic chemicals. Hydrophobic (insoluble) organic chemicals with low molecular weight will adsorb to this soil in small amounts. Hydrophobic chemicals with high molecular weight will adsorb in moderate amounts (Cline & Brown, 1989). These soils have a low capacity to hold contaminants in the grain interstices due to low capillary action. Contaminants that are held in these soils adhere to the grains themselves in dry soils and are forced into the smaller pore spaces in wet soils (Schwille, 1988).

**Where to sample:** Samples should be placed at regular intervals along the base and sidewalls of the excavation being sure that samples are located where the source was removed. In these soils, the capillary force is low enough to ignore its effects in transporting contaminants lateral to gravity. Therefore, sidewall samples should be located near the excavation floor. This is especially true for low surface tension products such as gasoline.

The limestone sand grains can act as a buffer to contaminants that cause pH changes (e.g., steel mill pickling acids). For these types of contaminants, the sampler should be on the lookout for intra-granular precipitates. These can appear as grain surface staining or make the soil appear clumpy or aggregated. Soils containing precipitates should be sampled.

-21-

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### ***Fine Sand and Silt***

These soils have strong capillary action due to the small inter-granular distances. A determination of the fluid surface tension of the spilled product is helpful. High surface tension aids in the ability of a substance to overcome gravity by capillary action. As before, higher molecular weight products can be expected to adsorb to the grains to a greater degree. This allows a product to move lateral to gravity and, to a degree, upward from the leak location. Low surface tension products, such as TCE (trichloroethene), are more likely to go straight down than oils in these kinds of soils. However, the hydraulic head (i.e., the amount of product in the original spill) must be substantial to force a dense non-aqueous phase liquid through a media with a hydraulic conductivity less than  $1 \times 10^{-3}$  cm/sec (Schwille, 1988).

**Where to sample:** Interfaces between fine sand layers with larger grains above should be sampled. When high surface tension contaminants are suspected, silt layers should be sampled.

### ***Clays***

~~Clay soils are very different from the sands and silts. Clays possess a net negative charge. This causes heavy metal cations (e.g.,  $\text{Cr}^{+6}$ ,  $\text{Cd}^{+2}$ ,  $\text{Pb}^{+2}$ ) to adsorb to the clay surface. In fact, this is true for any positive ionizable substance. Clays also have a much greater secondary porosity than primary (primary porosity is the space between the soil particles; secondary porosity is the space between fractures, bedding planes, and soil structures). As a result, spills in clay soils tend to follow preferred pathways. Clays will often show signs of shrinkage cracks or fractures that will allow contaminants to migrate in what would otherwise be considered a "tight" soil in a lab analysis of permeability. Signs of fracturing include "patterned" mottling. This is where the Fe (and also Mn) will be oxidized to a red, yellow, or reddish brown color along the crack while the matrix remains the reduced blue/gray color (Lindsay, 1979).~~

~~**Where to sample:** It is very important to take clay soil samples from fractures. The fractures are the avenue of travel for contaminants in clay soils. Clay soils may also have sand lenses which should always be sampled. Sand lenses in clays tend to collect fluids. As such, they may harbor contaminants.~~

### ~~**Bedrock**~~

~~Excavations in bedrock present difficult problems. Unlike clay, some bedrock formations have substantial primary porosity as well as secondary porosity. In Michigan, these are sandstones, conglomerates, and brecciated/coarse grained limestones. Examples of bedrock in Michigan with low primary porosity are fine grained limestones, shale, and crystalline metamorphic rocks (e.g., gneiss). If the sampler is unaware of the type of bedrock that is in an excavation, a geologist must be consulted.~~

~~**Where to sample:** Excavations in areas of bedrock with significant primary porosity must be sampled in both the fractures and the matrix. Bedrock without primary porosity should have sampling predominantly in the fractures as in the clay situation. Weathered zones in bedrock will hold contaminants better than unweathered zones. This is due to the increased number of adsorption sites available in weathered rock.~~

~~-22-~~

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## ~~**ATTACHMENT 2**~~

### ~~**SAMPLING PROTOCOL FOR CHARACTERIZING WASTE/TREATMENT LEVELS:**~~

#### ~~**STRATEGIES FOR EVALUATING TREATED SOILS AND WASTE MATERIALS**~~

~~When obtaining samples to characterize a treated soil or waste material, it is important to insure that the analytical results obtained will provide an accurate estimation of the nature of the entire area/volume under consideration. The location and number of samples to be taken at a particular site depend on many factors: the degree of accuracy desired, the spatial and temporal variability of the media to be sampled, and the costs involved. An important objective in any sampling program is to obtain the most accurate data possible while minimizing the associated costs. One method to accomplish this goal is to use statistically valid sampling strategies. The appropriate sample number can be estimated and the sampling locations can be chosen without bias.~~

Attachment 2 provides information on the methods used to obtain accurate data while minimizing the costs. The attachments include a discussion of three statistical sampling strategies and methods to determine the appropriate grid size for the area under investigation. If several areas on a site are under investigation, it may be advisable to grid them separately. This is especially true if information does not exist to indicate that the areas contain similar constituents or that they were placed at the same time period.

Information is also supplied on the statistical evaluation of the resultant analytical data. A minimum of 12 samples or 25%, whichever is greater, of the total grid stations should be sampled and analyzed initially to allow a large enough data pool for the statistical analysis. Extra samples should be taken and kept under proper chain of custody and handling procedures at the time of initial sampling. If the statistical analysis indicates that two or three more samples are needed, an additional trip to the field may not be necessary. This may also avoid the need to reestablish the grid pattern at a later date.

For further discussion on sampling strategies and sample collection methods, see "Test Methods for Evaluating Solid Waste," SW846 Volume II: Field Methods, November 1986, Third Edition, US EPA.

-23-

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## STATISTICAL SAMPLING STRATEGIES

Statistical sampling strategies can often produce increased data accuracy while eliminating sampler bias. Random sampling is based on the theory of random chance probabilities in order to choose the most representative sample. Knowledge of the waste distribution is not necessary. The error in data accuracy of a random sampling scheme can be objectively measured since the probability of choosing each sampling point is known. A random numbers table (attached) or a random numbers generator should be used to select the sampling locations eliminating bias by the sample collector.

Several statistical sampling strategies are available to produce an unbiased, representative sampling program. The principles behind three of these and the situations for which they are best suited are provided below. To achieve true random sampling, composite sampling is not acceptable.

1. **Simple Random** is a method that requires little or no prior knowledge of material distribution. It relies on random chance probability theory where each sampling location has an equal and known probability of being selected. In this way, sampling error can be accurately estimated. Usually, the area of interest is sectioned into a two or three dimensional grid pattern and random coordinates are chosen for sampling.

2. **Systematic random** is an extension of simple random sampling that may produce a more efficient sampling survey. It can be more efficient by reducing the sampling error while maintaining the sample number, or by reducing the number of samples needed to achieve a specified sampling error, or by reducing the cost of collection. This method also requires little or no knowledge about the waste distribution, but bias and imprecision can be introduced if unseen trends or cycles exist. Two methods used to select sample locations

under this method follow:

- A) randomly selecting a transect or transects and sampling at preselected intervals
- B) preselecting both the transect or transects and the sampling interval and starting from a randomly selected point.

3. **Stratified random sampling** requires some knowledge about the waste distribution. When stratification is known or suspected, sampling efficiency can be improved by dividing the material into strata that are more homogeneous than the total area. Simple random sampling techniques can then be used to sample each stratum independently. Each stratum is divided into a grid pattern and the sampling points are selected randomly. If the area is vertically stratified, the sampling points in each stratum are selected randomly and then selected depths are sampled. If the area is horizontally stratified, the sampling points within each stratum are selected randomly, but the total depth is sampled. An analysis of variance (ANOVA) should be done on the analytical results to determine if the strata differ significantly. This is done to assure that the use of stratified random sampling was statistically valid. When the volume of the strata differ or the number of samples within each strata differs, the results must be weighed appropriately to avoid bias.

-24-

## **RANDOM NUMBERS TABLE**

### **HOW TO USE THE RANDOM NUMBERS TABLE**

1. Determine the number of samples you need to take. Identify the number of digits necessary to cover the sample population (e.g., for a sample population of 55, two digits are necessary to cover the selected grid stations 01 through 55).
2. Using the random numbers table, choose any number as a starting point.
3. From this starting point number, go in any direction and continue in the same direction and pattern sequence until you have selected the predetermined number of samples with no repetitions. Numbers larger than the population size are ineligible (e.g., numbers greater than 55 in the example are ineligible).

Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)	
1	10480	15011	01536	02011	81647	91646	69179	14194	62590	36207	
2	22368	46573	25595	85393	30995	89198	27982	53402	93965	34095	
3	24130	48360	22527	97265	76393	64809	15179	24830	49340	32081	
4	42167	93093	06243	61680	07856	16376	39440	53537	71341	57004	
5	37570	39975	81837	16656	06121	91782	60468	81305	49684	60672	
6	77921	06907	11008	42751	27756	53498	18602	70659	90655	15053	

7	99562	72905	56420	69994	98872	31016	71194	18738	44013	48840
8	96301	91977	05463	07972	18876	20922	94595	56869	69014	60045
9	89579	14342	63661	10281	17453	18103	57740	84378	25331	12566
10	85475	36857	43342	53988	53060	59533	38867	62300	08158	17983
11	28918	69578	88231	33276	70997	79936	56865	05859	90106	31595
12	63553	40961	48235	03427	49626	69445	18663	72695	52180	20847
13	09429	93969	52636	92737	88974	33488	36320	17617	30015	08272
14	10365	61129	87529	85689	48237	52267	67689	93394	01511	26358
15	07119	97336	71048	08178	77233	13916	47564	81056	97735	85977
16	51085	12765	51821	51259	77452	16308	60756	92144	49442	53900
17	02368	21382	52404	60268	89368	19885	55322	44819	01188	65255
18	01011	54092	33362	94904	31273	04146	18594	29852	71585	85030
19	52162	53916	46369	58586	23216	14513	83149	98736	23495	64350
20	07056	97628	33787	09998	42698	06691	76988	13602	51851	46104
21	48663	91245	85828	14346	09172	30168	90229	04734	59193	22178
22	54164	58492	22421	74103	47070	25306	76468	26384	58151	06646
23	32639	32363	05597	24200	13363	38005	94342	28728	35806	06912
24	29334	27001	87637	87308	58731	00256	45834	15398	46557	41135
25	02488	33062	28834	07351	19731	92420	60952	61280	50001	67658
Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
26	81525	72295	04839	96423	24878	82651	66566	14778	76797	14780
27	29676	20591	68086	26432	46901	20849	89768	81536	86645	12659
28	00742	57392	39064	66432	84673	40027	32832	61362	98947	96067
29	05366	04213	25669	26422	44407	44048	37937	63904	45766	66134
30	91921	26418	64117	94305	26766	25940	39972	22209	71500	64568
31	00582	04711	87917	77341	42206	35126	74087	99547	81817	42607
32	00725	69884	62797	56170	86324	88072	76222	36086	84637	93161
33	69011	65797	95876	55293	18988	27354	26575	08625	40801	59920
34	25976	57948	29888	88604	67917	48708	18912	82271	65424	69774
35	09763	83473	73577	12908	30883	18317	28290	35797	05998	41688
36	91567	42595	27958	30134	04024	86385	29880	99730	55536	84855
37	17955	56349	90999	49127	20044	59931	06115	20542	18059	02008
38	46503	18584	18845	49618	02304	51038	20655	58727	28168	15475
39	92157	89634	94824	78171	84610	82834	09922	25417	44137	48413
40	14577	62765	35605	81263	39667	47358	56873	56307	61607	49518
41	98427	07523	33362	64270	01638	92477	66969	98420	04880	45585
42	34914	63976	88720	82765	34476	17032	87589	40836	32427	70002
43	70060	28277	39475	46473	23219	53416	94970	25832	69975	94884
44	53976	54914	06990	67245	68350	82948	11398	42878	80287	88267
45	76072	29515	40980	07391	58745	25774	22987	80059	39911	96189
46	90725	52210	83974	29992	65831	38857	50490	83765	55657	14361
47	64364	67412	33339	31926	14883	24413	59744	92351	97473	89286
48	08962	00358	31662	25388	61642	34072	81249	35648	56891	69352
49	95012	68379	93526	70765	10593	04542	76463	54328	02349	17247
50	15664	10493	20492	38391	91132	21999	59516	81652	27195	48223
51	16408	81899	04153	53381	79401	21438	83035	92350	36693	31238
52	18629	81953	05520	91962	04739	13092	97662	24822	94730	06496
53	73115	35101	47498	87637	99016	71060	88824	71013	18735	20286
54	57491	16703	23167	49323	45021	33132	12544	41035	80780	45393
55	30405	83946	23792	14422	15059	45799	22716	19792	09983	74353
56	16631	35006	85900	98275	32388	52390	16815	69298	82732	38480
57	96773	20206	42559	78985	05300	22164	24369	54224	35083	19687
58	38935	64202	14349	82674	66523	44133	00697	35552	35970	19124
59	31624	76384	17403	53363	44167	64486	64758	75366	76554	31601

60	78919	19474	23632	27889	47914	02584	37680	20801	72152	39339
61	03931	33309	57047	74211	63445	17361	62825	39908	05607	91284
62	74426	33278	43972	10119	89917	15665	52872	73823	73144	88662
63	09066	00903	20795	95452	92648	45454	09552	88815	16553	51125
64	42238	12426	87025	14267	20979	04508	64535	31355	86064	29472
65	16153	08002	26504	41744	81959	65642	74240	56302	00033	67107
66	21457	40742	29820	96783	29400	21840	15035	34537	33310	06116
67	21581	57802	02050	89728	17937	37621	47075	42080	97403	48626
68	55612	78095	83197	33732	05810	24813	86902	60397	16489	03264
69	44657	66999	99324	51281	84463	60563	79312	93454	68876	25471
70	91340	84979	46949	81973	37949	61023	43997	15263	80644	43942
71	91227	21199	31935	27022	84067	05462	35216	14486	29891	68607
72	50001	38140	66321	19924	72163	09538	12151	06878	91903	18749
73	65390	05224	72958	28609	81406	39147	25549	48542	42627	45233
74	27504	96131	83944	41575	10573	08619	64482	73923	36152	05184
75	37169	94851	39117	89632	00959	16487	65536	49071	39782	17095
Line/Col.	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)	(10)
76	11508	70225	51111	38351	19444	66499	71945	05422	13442	78675
77	37449	30362	06694	54690	04052	53115	62757	95348	78662	11163
78	46515	70331	85922	38329	57015	15765	97161	17869	45349	61796
79	30986	81223	42416	58353	21532	30502	32305	86482	05174	07901
80	63798	64995	46583	09765	44160	78128	83991	42865	92520	83531
81	82486	84846	99254	67632	43218	50076	21361	64816	51202	88124
82	21885	32906	92431	09060	64297	51674	64126	62570	26123	05155
83	60336	98782	07408	53458	13564	59089	26445	29789	85205	41001
84	43937	46891	24010	25560	86355	33941	25786	54990	71899	15475
85	97656	63175	89303	16275	07100	92063	21942	18611	47348	20203
86	03299	01221	05418	38982	55758	92237	26759	86367	21216	98442
87	79626	06486	03574	17668	07785	76020	79924	25651	83325	88428
88	85636	68335	47539	03129	65651	11977	02510	26113	99447	68645
89	18039	14367	61337	06177	12143	46609	32989	74014	64708	00533
90	08362	15656	60627	36478	65648	16764	53412	09013	07832	41574
91	79556	29068	04142	16268	15387	12856	66227	38358	22478	73373
92	92608	82674	27072	32534	17075	27698	98204	63863	11951	34648
93	23982	25835	40055	67006	12293	02753	14827	22235	35071	99704
94	09915	96306	05908	97901	28395	14186	00821	80703	70426	75647
95	50937	33300	26695	62247	69927	76123	50842	43834	86654	70959
96	42488	78077	69882	61657	34136	79180	97526	43092	04098	73571
97	46764	86273	63003	93017	31204	36692	40202	35275	57306	55543
98	03237	45430	55417	63282	90816	17349	88298	90183	36600	78406
99	86591	81482	52667	61583	14972	90053	89534	76036	49199	43716
100	38534	01715	94964	87288	65680	43772	39560	12918	86537	62738
101	13284	16834	74151	92027	24670	36665	00770	22878	02179	51602
102	21224	00370	30420	03883	96648	89428	41583	17564	27395	63904
103	99052	47887	81085	64933	66279	80432	65793	83287	34142	13241
104	00199	50993	98603	38452	87890	94624	69721	57484	67501	77638
105	60578	06483	28733	37867	07936	98710	98539	27186	31237	80612
106	91240	18312	17441	01929	18163	69201	31211	54288	39296	37318
107	97458	14229	12063	59611	32249	90466	33216	19358	02591	54263
108	35249	38646	34475	72417	60514	69257	12489	51924	86871	92446
109	38980	46600	11759	11900	46743	27860	77940	39298	97838	95145
110	10750	52745	38749	87365	58959	53731	89295	59062	39404	13198
111	36247	27850	73958	20673	37800	63835	71051	84724	52492	22342
112	70994	66986	99744	72438	01174	42159	11392	20724	54322	36923

113	99638	94702	11463	18148	81386	80431	90628	52506	02016	85151
114	72055	15774	43857	99805	10419	76939	25993	03544	21560	83471
115	24038	65541	85788	55835	38835	59399	13790	35112	01324	39520
116	74976	14631	35908	28221	39470	91548	12854	30166	09073	75887
117	35553	71628	70189	26436	63407	91178	90348	55359	80392	41012
118	35676	12797	51434	82976	42010	26344	92920	92155	58807	54644
119	74815	67523	72985	23183	02446	63594	98924	20633	58842	85961
120	45246	88048	65173	50989	91060	89894	36063	32819	68559	99221
121	76509	47069	86378	41797	11910	49672	88575	97966	32466	10083
122	19689	90332	04315	21358	97248	11188	39062	63312	52496	07349
123	42751	35318	97513	61537	54955	08159	00337	80778	27507	95478
124	11946	22681	45045	13964	57517	59419	58045	44067	58716	58840
125	96518	48688	20996	11090	48396	57177	83867	86464	14342	21545

-27-

### SAMPLING GRIDS

1. A grid system should be established over the specified area (sidewalls and base). Grid point representation should be proportioned to the size of the area. It is recommended that one of the following equations be used to determine grid intervals for stationing.

$$\text{small site: } (A/\pi)^{1/2} / 2 = GI$$

$$\text{medium site: } (A/\pi)^{1/2} / 4 = GI$$

$$\text{large site: } ((A*\pi) / SF)^{1/2} = GI$$

WHERE: A = area to be grid (square feet)

GI = grid interval

SF = Site Factor, length of area to be gridded (unitless)

pi = 3.14159

It appears that there are logical size ranges of sites to which the grid equations apply:

A) small: up to 0.25 acre

B) medium: 0.25-3.0 acres

C) large: 3.0 acres and greater

To simplify this application, use the following chart based on an average size range of sites (1 acre = 43,560 square feet). The approximate grid ranges are provided as a quick check on numbers generated for specific sites using the above formulas:

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<del>Site Acreage*</del>	<del>Square Feet*</del>	<del>Grid Interval Ranges</del>
<del>up to 0.25 (small)</del>	<del>up to 10,890</del>	<del>See Part 1</del>
<del>0.25-3.00 (medium)</del>	<del>10,890-130,680</del>	<del>15-50 feet</del>
<del>3.0 and over (large)</del>	<del>130,680 +</del>	<del>30 feet plus</del>

~~\* Site acreage, square footage, is total area of sidewalls and base of excavati~~

---

~~2. After the grid interval is calculated, it is recommended that a sealed grid overlay be made to superimpose on the area under consideration. Some specified point (usually the southwest corner) should be designated as the 0,0 coordinate. The grid can then be adjusted to maximize sampling coverage. Some grid adjustment may be necessary for unusually shaped areas.~~

~~-28-~~

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## ~~STATISTICAL EVALUATION~~

### ~~WASTE/TREATMENT CHARACTERIZATION SAMPLINGS~~

~~Following is a step by step description of the approach used to calculate confidence limits based on the analytical data derived from the preliminary samples.~~

- ~~1. Calculate a preliminary estimate of the mean,  $\bar{X}$~~

$$\bar{X} = (X_1 + X_2 + X_3 + \dots + X_n) / n$$

~~where:~~

~~n = number of measurements  
 $\bar{X}$  = variable concentration  
 $X_i$  = individual measurements~~

- ~~2. Calculate a preliminary estimate of the variance ( $S^2$ ) and the standard deviation ( $S$ ). Standard deviation is a function of both sampling variability and measurement variability.~~

$$S^2 = [(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \dots + (X_n - \bar{X})^2] / (n - 1)$$

$$S = (S^2)^{1/2}$$

- ~~3. Calculate the standard error of the mean ( $S_x$ ). Standard error is inversely proportional to the square root of the number of samples (increasing n from 4 to 16 reduces  $S_x$  by 50%).~~



$$Sx = S / (n)^{1/2}$$

4. Since the concern is only whether the upper limit of a confidence interval is below or above the regulatory threshold, the lower confidence limit (LCL) need not be considered. The upper confidence limit (UCL) can be calculated using the onetailed (onesided) t values with n-1 degrees of freedom derived from a table of the Student's t distribution. Where only small sized statistical samples are involved (n<30), the normal or Gaussian distribution is not accurate, and the t distribution must be used.

-29-

5. The 95% UCL is calculated by using the following formula and substituting the values determined above plus the appropriate t value obtained from the t table.

$$UCL = X + [t_{0.95,(n-1)}] * Sx$$

The term in brackets indicates a one-tailed t-test at n-1 degrees of freedom. See the t-distribution table in Attachment 2.

The UCL number resulting from this formula will indicate with a 95% probability that it is either above or below the regulatory threshold (RT) developed for the constituent being subjected to the test. If a compound does not have a specified RT, then the UCL is compared to whatever concentration is of concern (i.e., a clean up level, action level, etc). Other confidence levels can be used, based on the specific sampling situation.

If the preliminary data indicate that more samples are needed to make a hazard determination, the Lambda relationship should be used. A step by step approach to calculating the appropriate sample size follows:

1. The appropriate number of samples to be collected can be estimated by use of the Lambda relationship and then consulting a table of values and their corresponding sample size number.

$$Lambda = (RT - X) / S$$

The lower the calculated value, the more samples are required to maintain a certain level of confidence. Also, as approaches RT, Lambda becomes smaller, and therefore a greater sample size is indicated for a certain level of confidence.

2. To obtain the appropriate sample size from the table of values, use the single sided value for to test at the desired significance level (for 5%, = 0.05).

3. Randomly collect any additional samples that may be needed using the same grid and random numbers sequence as the first sampling. All field and laboratory procedures should be kept as consistent as possible to lower the amount of variability in the data.

4. Use all data values to calculate new X, S, and Sx.

-30-

5. If the new  $X \geq RT$ , then the contaminant is present at an unacceptable concentration and the study would be complete.

6. If  $X < RT$  and  $X > S^2$ , calculate  $C$  (the criterion for determining if contamination is present at hazardous concentration). If  $X = S^2$  or  $X < S^2$ , the data must be transformed prior to calculating  $C$ . Using the new data,  $C$  is calculated by the formula:

$$C = (RT - X) / Sx$$

7. Compare the calculated  $C$  value to the two-tailed  $t$  value for the level of significance desired. The two-tailed  $t$ -value is used because both the possibility that  $C$  is  $> t$  or that  $C$  is  $< t$  must be checked.

Use  $t_{0.95}$  and  $df$  (degrees of freedom) =  $n - 1$ .

8. If  $C > t$  value, the contaminant is present at unacceptable concentrations and the study would be over. If  $C < t$  value, reestimate the total number of additional samples to be collected by deriving a new  $\Lambda$ . Use the newly calculated values of  $X$  and  $S$ .

9. If this new number of samples is not more than 20% greater than the last set collected, there is little chance that additional samples would decrease  $Sx$  and result in the material being considered unacceptable. Therefore, the study would be complete.

-31-

## EXAMPLE

### CALCULATION OF CONFIDENCE LIMITS AND LAMDA CALCULATION

#### *Problem 1: STATISTICAL SAMPLING*

A metal plating factory has been discharging process wastewaters into a large nearby swampy area for several years. This swampy area drains into a small river. The discharged wastewaters are known to be contaminated with very low levels of cadmium and chromium (i.e., the levels in the wastewater are below the facilities NPDES permit limitations). However, it has been suspected that the sediments in this swampy area may contain high levels of cadmium and chromium. Three preliminary sediment samples were taken with a Ponar dredge and analyzed to determine whether or not these sediments were contaminated with hazardous levels of these two metals. In 40 CFR 261.24, it states that a waste is hazardous under the characteristic of EP toxicity if it contains cadmium at a level  $\geq 1.0$  mg/l or chromium at a level  $\geq 5.0$  mg/l. The analysis of the three preliminary samples indicated a mean cadmium concentration of 0.37 mg/l (3 samples at 0.25, 0.51, and 0.35 mg/l) and a mean chromium concentration of 4.66 mg/l (3 samples at 4.93, 4.21, and 4.84 mg/l). Based on this analytical data, the cadmium level is well below the regulatory threshold (RT), but

the chromium level closely approaches its RT. Because large legal or monetary losses may be incurred if the sediments are declared hazardous, the analytical data must be sound and a high degree of confidence is necessary in any decision made.

QUESTIONS: Given the above scenario, answer the following questions and calculate the appropriate answers.

1. Based on the chromium data supplied

Calculate  $S^2$ ,  $S$ ,  $S_x$

Calculate the 95% UCL

With what degree of confidence can it be stated that the chromium concentration does not exceed the RT?

2. If more samples are deemed necessary, determine how many

Calculate the Lambda value

Calculate the appropriate number of additional samples using  $\alpha=0.05$  and  $\beta=0.05$

-32-

## PROBLEM 1 ANSWER SHEET

Given three samples with chromium concentrations of 4.93, 4.21, and 4.84 mg/l and

$$\bar{X} = 4.66 \text{ mg/l}$$

(1a) Calculate  $S^2$

$$\begin{aligned} S^2 &= [(X_1 - \bar{X})^2 + (X_2 - \bar{X})^2 + \dots + (X_n - \bar{X})^2] / (n - 1) \\ &= [(4.93 - 4.66)^2 + (4.21 - 4.66)^2 + (4.84 - 4.66)^2] / 2 \\ &= 0.15 \end{aligned}$$

Calculate  $S$

$$S = (S^2)^{1/2} = (0.15)^{1/2} = 0.39$$

Calculate  $S_x$

$$S_x = S/(n)^{1/2} = 0.39/(3)^{1/2} = 0.23$$

---

~~(1b) Calculate the 95% UCL~~

$$\begin{aligned} 95\% \text{ UCL} &= X + [t_{0.95,(n-1)}] * Sx \\ &= 4.66 + (2.920) * (0.23) \\ &= 5.33 \end{aligned}$$

---

~~(1c)~~

$$\begin{aligned} 90\% \text{ UCL} &= X + [t_{0.90,(n-1)}] * Sx \\ &= 4.66 + (1.886) * (0.23) \\ &= 5.09 \\ 80\% \text{ UCL} &= X + [t_{0.80,(n-1)}] * Sx \\ &= 4.66 + (1.061) * (0.23) \\ &= 4.90 \end{aligned}$$

~~-33-~~

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~~The preceding two calculations indicate that it can be stated with somewhere between 80% and 90% confidence that the chromium concentration does not exceed the RT. This degree of confidence may not be sufficient to meet the needs of the sampling plan. Therefore, more samples may need to be taken.~~

~~2a. Calculate the Lambda value~~

$$\begin{aligned} \text{Lambda} &= (RT - X) / S \\ &= (5.0 - 4.66) / 0.39 = 0.87 \end{aligned}$$

~~2b. Calculate the number of additional samples~~

~~Using Attachment 2, Number of Observations for t Test of Mean, page 36 of this Guidance Document, using a single-sided test with  $\alpha=0.05$  and  $\beta=0.05$ , approximately 15 to 17 total samples need to be collected. Therefore, based on the three preliminary samples that were collected, an additional 13 samples need to be taken.~~

~~-34-~~**Cumulative t Distribution**

<del>one tailed</del>	<del>0.550</del>	<del>0.750</del>	<del>0.080</del>	<del>0.900</del>	<del>0.950</del>	<del>0.975</del>	<del>0.990</del>	<del>_____</del>
<del>two tailed</del>	<del>0.100</del>	<del>0.500</del>	<del>0.600</del>	<del>0.800</del>	<del>0.900</del>	<del>0.950</del>	<del>0.980</del>	<del>_____</del>
1	0.158	1.000	1.376	3.078	6.314	12.706	31.82	_____
2	0.142	0.816	1.061	1.886	2.920	4.303	6.925	_____
3	0.137	0.765	0.978	1.638	2.353	3.182	4.541	_____
4	0.134	0.741	0.941	1.533	2.132	2.776	3.747	_____
5	0.132	0.727	0.920	1.476	2.015	2.571	3.365	_____
6	0.131	0.718	0.906	1.440	1.943	2.447	3.143	_____
7	0.130	0.711	0.896	1.415	1.895	2.365	2.998	_____
8	0.130	0.706	0.889	1.397	1.860	2.306	2.896	_____
9	0.129	0.703	0.883	1.383	1.833	2.262	2.821	_____
10	0.129	0.700	0.879	1.372	1.812	2.228	2.764	_____
11	0.129	0.697	0.876	1.363	1.796	2.201	2.718	_____
12	0.128	0.695	0.873	1.356	1.782	2.179	2.681	_____
13	0.128	0.694	0.870	1.350	1.771	2.160	2.650	_____
14	0.128	0.692	0.868	1.345	1.761	2.145	2.624	_____
15	0.128	0.691	0.866	1.341	1.753	2.131	2.602	_____
16	0.128	0.690	0.865	1.337	1.746	2.120	2.583	_____
17	0.128	0.689	0.863	1.333	1.740	2.110	2.567	_____
18	0.127	0.688	0.862	1.330	1.734	2.101	2.552	_____
19	0.127	0.688	0.861	1.328	1.729	2.093	2.539	_____
20	0.127	0.687	0.860	1.325	1.725	2.086	2.528	_____
21	0.127	0.686	0.859	1.323	1.721	2.080	2.518	_____
22	0.127	0.686	0.858	1.321	1.717	2.074	2.508	_____
23	0.127	0.685	0.858	1.319	1.714	2.069	2.500	_____
24	0.127	0.685	0.857	1.318	1.711	2.064	2.492	_____
25	0.127	0.684	0.856	1.316	1.708	2.060	2.485	_____
26	0.127	0.684	0.856	1.315	1.706	2.056	2.479	_____
27	0.127	0.684	0.855	1.314	1.703	2.052	2.473	_____
28	0.127	0.683	0.855	1.313	1.701	2.048	2.467	_____
29	0.127	0.683	0.854	1.311	1.699	2.045	2.462	_____
30	0.127	0.683	0.854	1.310	1.697	2.042	2.457	_____
40	0.126	0.681	0.851	1.303	1.684	2.021	2.423	_____
60	0.126	0.679	0.848	1.296	1.671	2.000	2.390	_____
120	0.126	0.677	0.845	1.289	1.658	1.980	2.358	_____
	0.126	0.674	0.842	1.282	1.645	1.960	2.326	_____

NOTE: For one tailed distributions  $\alpha/2 = 1$  p  
 For two tailed distributions  $\alpha = 1$  p

~~-35-~~

**NUMBER OF OBSERVATIONS FOR t TEST OF MEAN**  
**Level for t Test**

Single sided ~~A=0.005~~  
 Double sided ~~A=0.01~~

~~A=0.01~~  
~~A=0.02~~

~~A=0.025~~  
~~A=0.05~~

~~B=0.01 0.05 0.1 0.2 0.5 0.01 0.05 0.1 0.2 0.5 0.01 0.05 0.1 0.2~~

~~LAMBDA~~

<del>0.15</del>															
<del>0.20</del>															
<del>0.25</del>															
<del>0.30</del>															
<del>0.35</del>															
<del>0.40</del>															
<del>0.45</del>															
<del>0.50</del>															
<del>0.55</del>															
<del>0.60</del>															
<del>0.65</del>															
<del>0.70</del>															
<del>0.75</del>															
<del>0.80</del>															
<del>0.85</del>															
<del>0.90</del>															
<del>0.95</del>															
<del>1.00</del>															
<del>1.1</del>															
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<del>1.8</del>															
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<del>2.3</del>															
<del>2.4</del>															
<del>2.5</del>															
<del>3.0</del>															
<del>3.5</del>															
<del>4.0</del>															

~~99% confidence~~~~-36-~~~~SAMPLING PROCESS STREAMS~~

~~Although sampling is generally thought to occur on a pile of material or over an area of treated soil, other schemes are possible. The most common instance is when the material is to be sampled at the point of generation. This is the preferred method, since it is most representative of the material under study. The lack of exposure to elements that might cause chemical degradation and/or leaching will result in material most indicative of actual conditions.~~

~~A sampling point along the material conveyor that can be fairly easily and safely reached should be chosen. It should be in an area where the entire belt can be accessed for sampling.~~

~~Under this scenario, a temporal, rather than a spatial, approach needs to be used.~~

~~Time stratum should be established over the course of the process day. Ideally, the entire active time of the line should be included in the sampling scheme. Once time strata are chosen, the random numbers table can be used to establish sampling times. For a four hour period, a point somewhere on the table would be chosen and every number greater than 0 but less than 240 would be selected until the number of samples for that strata were obtained. The number would relate to time in minutes. This would be added to the starting time for that strata to determine the time of sampling.~~

~~If the time strata chosen are of unequal lengths, the number of samples chosen from any one strata should reflect the percentage contribution that strata makes to the time frame as a whole. For example, if for a 24 hour operating time, strata 1 is 4 hours and strata 2 is 8 hours, strata 2 should have twice as many samples as strata 1.~~

~~When the appropriate sampling time arrives, the material from the conveyor belt point that had been identified would be removed. This material should be well mixed and a subsample taken for inclusion in the jar for lab analysis. An example of the use of this protocol is attached.~~

~~-37-~~

#### ~~RANDOM TIME WASTE SAMPLING EXAMPLE~~

	<del>Sampling Point</del>	<del>Random Minute</del>	<del>Time</del>
<del>Stratum #1</del>			
<del>6:00 to 8:00 hours</del>	<del>1</del>	<del>28</del>	<del>6:28</del>
	<del>2</del>	<del>62</del>	<del>7:02</del>
	<del>3</del>	<del>99</del>	<del>7:39</del>
	<del>4</del>	<del>112</del>	<del>7:52</del>
<del>Stratum #2</del>			
<del>8:00 to 20:00 hours</del>	<del>1</del>	<del>11</del>	<del>8:11</del>
	<del>2</del>	<del>107</del>	<del>9:47</del>
	<del>3</del>	<del>156</del>	<del>10:36</del>
	<del>4</del>	<del>173</del>	<del>10:53</del>
	<del>5</del>	<del>296</del>	<del>12:56</del>
	<del>6</del>	<del>313</del>	<del>13:13</del>
	<del>7</del>	<del>398</del>	<del>14:38</del>
	<del>8</del>	<del>497</del>	<del>16:17</del>
	<del>9</del>	<del>555</del>	<del>17:15</del>
	<del>10</del>	<del>600</del>	<del>18:00</del>
	<del>11</del>	<del>637</del>	<del>18:37</del>
	<del>12</del>	<del>706</del>	<del>19:46</del>
<del>Stratum #3</del>			
<del>20:00 to 22:00 hours</del>	<del>1</del>	<del>13</del>	<del>20:13</del>
	<del>2</del>	<del>52</del>	<del>20:52</del>
	<del>3</del>	<del>88</del>	<del>21:28</del>
	<del>4</del>	<del>108</del>	<del>21:48</del>
<del>Stratum #4</del>			

<del>8:00 to 20:00 hours</del>	<del>1</del>	<del>48</del>	<del>22:48</del>
	<del>2</del>	<del>113</del>	<del>23:53</del>
	<del>3</del>	<del>153</del>	<del>24:33</del>
	<del>4</del>	<del>189</del>	<del>1:09</del>
	<del>5</del>	<del>227</del>	<del>1:47</del>
	<del>6</del>	<del>290</del>	<del>2:49</del>
	<del>7</del>	<del>314</del>	<del>3:14</del>
	<del>8</del>	<del>474</del>	<del>5:44</del>

~~-38-~~

### ~~ATTACHMENT 3~~

#### ~~TOLERANCE FACTORS (K)~~

~~TOLERANCE FACTORS (K) FOR ONE SIDED NORMAL TOLERANCE INTERVALS WITH PROBABILITY  
Y = 0.95 AND COVERAGE P = 95%~~

<del>n</del>	<del>K</del>	<del>n</del>	<del>K</del>
<del>3</del>	<del>7.655</del>	<del>75</del>	<del>1.972</del>
<del>4</del>	<del>5.145</del>	<del>100</del>	<del>1.924</del>
<del>5</del>	<del>4.202</del>	<del>125</del>	<del>1.891</del>
<del>6</del>	<del>3.707</del>	<del>150</del>	<del>1.868</del>
<del>7</del>	<del>3.399</del>	<del>175</del>	<del>1.850</del>
<del>8</del>	<del>3.188</del>	<del>200</del>	<del>1.836</del>
<del>9</del>	<del>3.031</del>	<del>225</del>	<del>1.824</del>
<del>10</del>	<del>2.911</del>	<del>250</del>	<del>1.814</del>
<del>11</del>	<del>2.815</del>	<del>275</del>	<del>1.806</del>
<del>12</del>	<del>2.736</del>	<del>300</del>	<del>1.799</del>
<del>13</del>	<del>2.670</del>	<del>325</del>	<del>1.792</del>
<del>14</del>	<del>2.614</del>	<del>350</del>	<del>1.787</del>
<del>15</del>	<del>2.566</del>	<del>375</del>	<del>1.782</del>
<del>16</del>	<del>2.523</del>	<del>400</del>	<del>1.777</del>
<del>17</del>	<del>2.486</del>	<del>425</del>	<del>1.773</del>
<del>18</del>	<del>2.453</del>	<del>450</del>	<del>1.769</del>
<del>19</del>	<del>2.423</del>	<del>475</del>	<del>1.766</del>
<del>20</del>	<del>2.396</del>	<del>500</del>	<del>1.763</del>
<del>21</del>	<del>2.371</del>	<del>525</del>	<del>1.760</del>
<del>22</del>	<del>2.350</del>	<del>550</del>	<del>1.757</del>
<del>23</del>	<del>2.329</del>	<del>575</del>	<del>1.754</del>
<del>24</del>	<del>2.309</del>	<del>600</del>	<del>1.752</del>
<del>25</del>	<del>2.292</del>	<del>625</del>	<del>1.750</del>
<del>30</del>	<del>2.220</del>	<del>650</del>	<del>1.748</del>
<del>35</del>	<del>2.166</del>	<del>675</del>	<del>1.746</del>
<del>40</del>	<del>2.126</del>	<del>700</del>	<del>1.744</del>
<del>45</del>	<del>2.092</del>	<del>725</del>	<del>1.742</del>
<del>50</del>	<del>2.065</del>	<del>750</del>	<del>1.740</del>
<del>55</del>	<del>2.036</del>	<del>775</del>	<del>1.739</del>
<del>60</del>	<del>2.017</del>	<del>800</del>	<del>1.737</del>
<del>65</del>	<del>2.000</del>	<del>825</del>	<del>0.736</del>
<del>70</del>	<del>1.986</del>	<del>850</del>	<del>1.734</del>
		<del>875</del>	<del>1.733</del>
		<del>900</del>	<del>1.732</del>
		<del>925</del>	<del>1.731</del>
		<del>950</del>	<del>1.729</del>
		<del>975</del>	<del>1.728</del>



~~1,000~~

~~1.727~~

~~SOURCE: FOR SAMPLE SIZES < 50: Lieberman, Gerald F. 1958. "Tables for One-sided Statistical Tolerance Limits." Industrial Quality Control. Vol. XIV, No. 10.~~

~~FOR SAMPLE SIZES > 50: K values were calculated from large sample approximation.~~

~~NTIS Document PB-89-151-047~~

~~-39-~~

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## ~~ATTACHMENT 4~~

### ~~WASTE MANAGEMENT DIVISION'S~~

#### ~~CLEAN CLOSURE CERTIFICATION CHECKLIST~~

~~This checklist was developed to review RCRA clean closures. Due to direct reference to 40 CFR, Part 264, Subpart G, by Act 64, Rule 613; Act 64 closures should also be evaluated by this checklist.~~

~~Documentation supporting the owners/operators and the independent registered professional engineer's certification can be requested under 40 CFR, 264.115 and 265.115 (as of October 29, 1986). The owner/operator must submit at least four copies of certification documentation.~~

~~The checklist identifies items recommended to properly evaluate a closure certification. These items are not "absolutes." Other information or substitutions may be provided which technically justify and certify a "clean closure."~~

~~This checklist can be used for land disposal, storage, and treatment facilities. Several of the items would not be required for storage and/or treatment facilities where testing was minimal. Items 1 through 5 would be required for all closures. Items 6 through 11 would be optional for storage and/or treatment facilities, dependent on extent of testing required. Land disposal facilities would require all items listed.~~

- ~~1. Manifests (or some type of manifest/waste removal summary) of where and how much waste was shipped.~~
- ~~2. Certification statement is needed by the owner/operator AND an independent registered engineer. All independent registered professional engineer certificates must have an original stamp on at least one copy.~~
- ~~3. Summary of decontamination procedures (pressure wash, Steam clean, etc.) and how the resultant waste water was disposed.~~
- ~~4. Summary analysis (include conditions of haul roads, time table, soil and groundwater~~

~~results, weather conditions, runoff controls, equipment decontamination, etc.).~~

~~5. Results of all tests used to determine clean closure (charts, tables, lab sheets).~~

~~6. Statistical comparisons on sampling results compared to cleanup criteria (this should include full computations on background and statistical analysis).~~

~~7. Sampling and analysis procedures (specify references).~~

~~8. Final depth and elevations of excavations of wastes and soils.~~

~~-40-~~

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~~9. Properly labeled and easily identified sampling locations and grid stations (map) including background stations.~~

~~10. Groundwater data (and statistical evaluation) used to determine if groundwater degradation has occurred (usually four sets of replicate analysis compared to sampling event after closure activities). Monitor well construction details and sampling and analysis procedures may be required if documentation is not in the file.~~

~~11. Summary of final restoration of excavated area... information on fill material used and/or future land use outline. If clean closure cannot be achieved (e.g., contaminated soils to water table and groundwater results show contamination). This summary item should be used to address the need for any post closure program and/or corrective action.~~

~~12. A copy of all field notes pertaining to these closure activities.~~

~~13. A copy of the approved closure plan and letter of closure plan approval.~~

~~-41-~~

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## **~~CITED REFERENCES~~**

~~Borden, R.C., Bedient, P.B., In-Situ Measurement of Adsorption and Biotransformation at a Hazardous Waste Site, Water Resources Bulletin, 23 (4), pp. 629-636, 1987~~

~~Cline, P.V., Brown S., Site Specific Evaluation of Contaminant Fate: Test Applications and Limitations, Superfund '89, Hazardous Materials Control Research Institute, Silver Springs, MD, 1989~~

~~Davis, J.C., Statistics and Data Analysis in Geology, 2nd ed., John Wiley and Sons, New York, 1986~~

~~Gilbert, R.O., Statistical Methods for Environmental Pollution Monitoring. Van Nostrand,~~

~~New York, 1987~~

~~Lindsay, W.L., Chemical Equilibria In Soils, John Wiley and Sons, New York, 1979~~

~~Schwille, F., Dense Non-Aqueous Phase Liquids In Porous and Fractured Media Model Experiments, Lewis Publishers, Boca Raton, LA, 1988~~

~~US EPA, Office of Policy, Planning, and Evaluation, Methods for Evaluating the Attainment of Cleanup Standards, Volume 1: Soils and Solid Media, EPA 230/02-89-042, Washington D.C., 1989~~

~~US EPA, Office of Solid Waste Management Division, Test Methods for Evaluating Solid Waste, SW-846 Volume II: Field Methods, US EPA, November 1986, Third Edition~~

~~US EPA, Office of Solid Waste Management Division, Statistical Analysis of Groundwater Monitoring Data at RCRA Facilities, Interim final Guidance, EPA, Washington D.C., April 1989.~~

~~US EPA, Office of Solid Waste Management Division, Statistical Analysis of Groundwater Monitoring data at RCRA Facilities, Addendum to Interim Final Guidance, EPA, Washington D.C., June 1992.~~

~~Wilson, J.T., Wilson B.H., Biotransformation of Trichloroethylene in Soil, Applied Environmental Microbiology, 49:242-243, 1985~~

~~WMD, DNR, Draft Guidance, How Clean is Clean, May 1988~~

~~ASTM STP-540, Sampling Standards and Homogeneity, Kennedy/Woodruff, Baltimore MD, 1973~~

~~ASTM STP-845, Statistics in the Environmental Sciences, Gentz/London editors, Baltimore MD, 1986~~

~~Underwood, E.E., Quantitative Stereology. Addison-Wesley, Reading Massachusetts, 1970~~

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~~Revised November 15, 2001~~

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